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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification <sup>5</sup> :</b> <b>A61K 7/50</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 94/16680</b> <b>(43) International Publication Date:</b> 4 August 1994 (04.08.94)
<b>(21) International Application Number:</b> PCT/EP94/00146 <b>(22) International Filing Date:</b> 19 January 1994 (19.01.94) <b>(30) Priority Data:</b> 9301270.6 22 January 1993 (22.01.93) GB <b>(71) Applicant (for AU BB CA GB IE LK MN MW NZ SD only):</b> UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB). <b>(71) Applicant (for all designated States except AU BB CA GB IE LK MN MW NZ SD):</b> UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). <b>(72) Inventors:</b> MOULD, Roy, Arthur, Stedman; 6 Moseley Road, Spital, Merseyside L63 9NT (GB). SALMON, Tom, Matthew, F.; Viale Risorgimento, 33/b, I-29100 Piacenza (IT). <b>(74) Agent:</b> BRYANT, Tracey; Unilever plc, Patent Division, Colworth House, Sharnbrook, Bedford MK44 1LQ (GB).		<b>(81) Designated States:</b> AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, ES, FI, GB, HU, JP, KP, KR, KZ, LK, LU, LV, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SK, UA, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> DILUTION-THICKENING, PERSONAL WASHING COMPOSITION  <b>(57) Abstract</b>  An aqueous dilution-thickening, concentrated personal washing composition comprises: a) 20-60 wt% surfactant other than soap or primary alcohol sulphate; and b) 1-20 wt% electrolyte, the ratio of electrolyte to surfactant being sufficient to form a lamellar phase. It has a viscosity which is low enough for processing, packaging and dispensing and thicken in use to a sufficiently high viscosity that does not run off the skin or hair when applied thereto.		

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DILUTION-THICKENING, PERSONAL WASHING COMPOSITION

5 The invention relates to dilution-thickening, personal washing compositions, suitable for washing of skin and/or hair.

10 Commercially available personal washing compositions, i.e. shower gels, facial cleansers and hair shampoos, generally comprise 15-20%wt surfactant. These compositions typically have a viscosity in the range 2-10 Pascal seconds, and become less viscous on dilution. At these lower viscosities, the composition tends to run off, or be washed rapidly off, body surfaces to which it is applied, leading to wastage of the product.

15 For reasons of economy of packaging, storage, distribution and portability, it is desirable to produce more concentrated products. At higher product concentrations, losses due to run-off become even more significant, it is therefore desirable to thicken the products so as to prevent or reduce run-off. However, thick products are more difficult to process, package and dispense.

25 In view of the above, products should have a viscosity which is low enough for processing, packaging and dispensing, but sufficiently high that the products do not run off the skin or hair when applied. In addition to this, products need to be stable over a range of temperatures, homogeneous and comprise formulations which can suffer small variations in raw material properties without loss of stability and performance. Furthermore, products should be mild to the skin when used.

35 Japanese patent application JP 63-270533 (Shiseido KK) discloses the formulation of facial cleansers which thicken on addition of a small quantity of water. The

compositions disclosed by way of example are based on around 40% of soaps or primary alcohol sulphates as anionic surfactant, water and 10-25% of a polyhydric alcohol, preferably propylene glycol as hydrotrope. In  
5 some instances, the examples require the use of triethanolamine as a solubiliser for the soap.

Soaps and primary alcohol sulphates are particularly harsh surfactants, and it is desirable to employ less harsh  
10 surfactants in personal washing compositions. For the purposes of the present application the term 'soaps' includes alkali metal or alkanol ammonium salts of aliphatic alkane or alkene monocarboxylic acids.

15 Many less harsh, i.e. more mild, surfactants are known, however, these surfactants, unlike fatty acid soaps, are impossible to obtain in a relatively anhydrous form in commercial quantities, and are generally supplied as an admixture with water, typically as 30-40%w/w solutions.  
20 The presence of excess water in the raw materials leads to formulation difficulties, particularly where the desired concentration levels are above 30% surfactant on product.

Moreover, the use of high levels of hydrotrope, such as  
25 the above-mentioned polyhydric alcohols is not desirable for reasons of cost and further limits the formulation space available.

In addition, triethanolamine (TEA) is not a preferred  
30 ingredient for personal washing compositions due to the potential for contaminants present in the TEA to form nitrosamines.

We have determined that stable dilution-thickening, and in  
35 particular concentrated, washing composition suitable for use on skin or hair can be manufactured using mild,

synthetic surfactants, electrolyte and water, without requiring the presence of triethanolamine or polyhydric alcohols.

5 Accordingly, the present invention provides an aqueous, stable dilution-thickening, concentrated, washing composition comprising:

- 10 a) 20-60wt% surfactant other than soap or primary alcohol sulphate, and,
- b) 1-20wt% electrolyte, the ratio of electrolyte to surfactant being sufficient to form a lamellar phase.

15 It is believed that the compositions of the prior art, i.e. known shower gels, are viscous due to the formation of a hexagonal or viscous rod phase in the products, due to the interaction of the lower levels of surfactant and electrolyte. Compositions of the present invention are  
20 selected such that a relatively low-viscosity, lamellar phase is formed in the undiluted products. These concentrated products can therefore be packaged and dispensed with ease. On addition of water, a viscous rod phase or hexagonal phase is formed, preventing or reducing  
25 run-off.

Typically, compositions according to the present invention comprise 30-60% surfactant. As mentioned above, conventional shower gels comprise up to around 25%  
30 surfactant. Products with higher levels of surfactant are desirable due to economies in packaging, storage and transportation.

Generally, the compositions according to the present  
35 invention are free of triethanolamine, hydrotropic

polyhydric alcohols and mixtures thereof. These components are undesirable for the reasons given above.

5 Preferably, the surfactant comprises at least one anionic surfactant.

Preferred levels of anionic surfactant are such that the anionic comprises 10-100wt% of the total surfactant system. Particularly preferred levels of anionic range  
10 from 50-90wt%, more preferably 70-90% of the total surfactant system.

Suitable anionic surfactants are selected from the group comprising: fatty acid ether sulphates, alpha-olefin  
15 sulphonates, fatty isethionates, alkyl glyceryl ether sulphonates, mono-alkyl glyceryl sulphates, alkyl sarcosinates, alkyl taurides, alkyl and dialkyl sulphosuccinates, alkyl phosphates, and mixtures thereof.

20 Preferred amongst the anionic surfactants are fatty acid ether sulphates, particularly lauryl ether sulphate salts such as sodium lauryl ether sulphate (SLES). Alternative, preferred anionic surfactants include alkyl sulphosuccinate and fatty isethionates.

25 As mentioned above, sodium lauryl ether sulphate (SLES) is particularly preferred as the anionic surfactant. SLES is a mixture of compounds having a mean chain length of from C<sub>10</sub>-C<sub>14</sub>, generally around C<sub>12</sub> and is commercially available in a concentrated form.

30 Preferably, compositions according to the present invention further comprise a synergistic mildness active (coactive).

35 More preferably, the synergistic mildness active is selected from the group consisting of anionic surfactants,

nonionic surfactants, amphoteric surfactants and mixtures thereof.

When present, the synergistic mildness active should be present at a level of at least 5%wt of the total active level as little or no synergy is noted at lower levels. Particularly useful compositions comprise 10-40%wt, preferably 15-30%wt, more preferably around 20%wt of synergistic mildness active on total actives.

10

Suitable nonionic surfactants include:- polyethoxylated alcohols, polyethoxylated alkyl phenols, alkyl polyglycosides, sorbitan esters, polysorbates, alkanolamides, poloxamers, and mixtures thereof.

15

Preferred amongst the nonionic surfactants are polyethoxylated alcohols, particularly C<sub>8</sub>-C<sub>18</sub> ethoxylates of 3-25 EO and alkylpolyglycosides. The most preferred ethoxylates have an average alkyl chain length of 12-16 carbons and an average ethoxylate content of 4-20 units.

20

Suitable amphoteric surfactants include:- amine oxides, aminimides, betaines, amido betaines and sulpho betaines, and mixtures thereof. Coco-amidopropyl betaines are particularly preferred due to their low potential nitrosamine-precursor content and mildness properties.

25

Suitable anionic co-actives include the sulphosuccinates, preferably the mono-alkyl sulphosuccinates with or without a linking group between the alkyl tail and the head group. Coco-alkylolamide sulphosuccinates are particularly preferred.

30

The most preferred synergistic mildness actives are the betaines, particularly coco-amidopropyl betaines.

35



Particularly preferred compositions according to the present invention comprise:

- 5 a) 30-60wt% surfactant, comprising a mixture of at least one betaine and at least one anionic surfactant other than soap or primary alcohol sulphate, in a ratio of 1:1 to 1:10, and,
- 10 b) 1-20wt% electrolyte, the ratio of electrolyte to surfactant being sufficient to form a lamellar phase.

Specific embodiments of the present invention comprise:

- 15 a) 30-60wt% surfactant, comprising a mixture of at least one betaine and a fatty acid ether sulphate salt, preferably in a ratio of 1:1 to 1:10 and,
- 20 b) 1-20wt% electrolyte, the ratio of electrolyte to surfactant being sufficient to form a lamellar phase.

20 More particularly, preferred embodiments of the invention comprise 40-60%wt of a surfactant system comprising 4-5 parts fatty acid ether sulphate in combination with 1-2 parts of a co-active selected from the group comprising,

25 coco-amido propyl, betaine, coco-alkylolamide sulphosuccinate, alkyl ethoxylated sulphosuccinate, 12-16 carbons, 4-20 EO alcohol ethoxylates, alkyl poly-glycoside and mixtures thereof.

30 A wide range of electrolytes are suitable. Preferred electrolytes are selected from the group comprising alkali metal or ammonium halides, particularly chlorides. Sodium chloride is the most preferred electrolyte, and is preferably present in a ratio to the surfactant of 1:10

35 salt:surfactant although the precise limits of the range

within which the lamellar phase will be formed will vary with the type of surfactant used.

5 Preferred compositions according to the present invention further comprise at least one skin or hair conditioning agent.

10 Preferred polymeric conditioning agents are the cationic, anionic, amphoteric, and the nonionic polymers used in the cosmetic field. Reduced skin irritation benefits as measured by patch testing of cationic and nonionic types of polymers are set out in "Polymer JR for Skin Care" Bulletin, by Union Carbide, 1977. The cationics are preferred over the others because they provide better skin  
15 feel benefits.

Suitable cationic polymers employed in this invention also provide a desirable silky, soft, smooth in-use feeling. The preferred level for this invention is 0.1-5% of the  
20 composition. There is reason to believe that the positively charged cationic polymers can bind with negatively charged sites on the skin to provide a soft skin feel after use. Not to be bound by any theory, it is believed that the greater the charge density of the  
25 cationic polymer, the more effective it is for skin feel benefits.

Typically, the cationic polymer is water soluble and has a cationic charge density (as defined in US patent 3580853)  
30 greater than 0.0001 in aqueous solution. Preferred levels of cationic polymers range from 0.1-5wt% as mentioned above, and are preferably present at a level of around 1wt%

35 Cationic charge density refers to the ratio of the number of positive charges on a monomeric unit of which the

polymer is comprised to the molecular weight of said monomeric unit.

5 Other suitable cationic polymers are copolymers of dimethyl-amino-ethyl-methacrylate and acrylamide and copolymers of dimethyl-diallyl-ammonium chloride and acrylamide in which the ratio of the cationic to neutral monomer units has been selected to give a copolymer having a cationic charge. Yet other suitable types of cationic  
10 polymers are the cationic starches, e.g., Sta-Lok<sup>®</sup>300 and 400 made by Staley, Inc.

Preferred silicones are essentially insoluble silicones, in order that they do not affect the rheology of the  
15 system by thinning the compositions.

Particularly preferred embodiments of the present invention comprise:

- 20 a) 30-50wt% surfactant, comprising a mixture of at least one betaine and a fatty acid ether sulphate salt, preferably in a ratio of 1:1 to 1:10 and,
- 25 b) 3-10wt% electrolyte, the ratio of electrolyte to surfactant being sufficient to form a lamellar phase, and,
- 30 c) 0.1-5wt% guar hydroxy propyl tri-methyl ammonium chloride polymer having a cationic charge density greater than 0.0001 in aqueous solution.

In addition to the above, compositions according to the present invention can comprise a non-hydrotrope polyol, preferably glycerol.

Minor components are inessential but typical components of personal washing compositions. These can be selected from one or more of opacifiers, preferably 0.2-2.0%wt; preservatives and perfumes, preferably 0.5-5 wt%.

5

Examples of preservatives include: ethanol, benzoic acid, sodium benzoate, sorbic acid, potassium sorbate, sodium propionate and the methyl, ethyl, propyl and butyl esters of p-hydroxybenzoic acid 2-bromo-2-nitropropane-1, 3-diol, phenoxyethanol, dibromodicyanobutane, formalin and Tricolsan. The amount of chemical preservative optionally to be incorporated in the composition according to the invention will generally be from 0.05 to 5%, preferably from 0.01 to 2% by weight, the amount chosen being sufficient to arrest microbial proliferation.

10

15

Optional further components include colouring agents, germicides and anti-oxidants.

20

It should be noted that these additional, technically inessential components can contain salts which may interfere with the overall electrolyte balance of the products and consequently the salt levels in these components should be taken into account.

25

In order that the invention may be further understood it will be illustrated hereafter by way of example.

30

EXAMPLES:

In the following examples the materials used are as listed below, all percentages are by weight:

- SLES: GENAPOL ZRO (RTM ex. Hoechst) sodium lauryl ether sulphate, average 3EO, 70% paste: anionic surfactant.
- 5 Sulp: REWOPOL SBC212P (RTM ex. REWO) coco-alkylolamide sulphosuccinate, 95-100% powder: anionic surfactant, used as co-active.
- 10 ESUL: REWOPOL SBFA30 (RTM ex. REWO) alkyl ethoxylated sulphosuccinate, 40% solution: anionic surfactant, used as co-active.
- 15 CAPB: REWOTERIC AMB14 (RTM ex. REWO) coco-amido propyl betaine, 30% aqueous solution: amphoteric surfactant, used as a co-active.
- 20 NONA: BRIJ-58 (RTM ex. ATLAS) C<sub>16</sub>-EO-20 average, alcohol ethoxylate, 100% waxy solid: nonionic surfactant, used as a co-active.
- 25 NONB: BRIJ-30 (RTM ex. ATLAS) C<sub>12</sub>-EO-4 average, alcohol ethoxylate, 100% liquid: nonionic surfactant, used as a co-active.
- APG: ORAMIX NS-10 (RTM ex. SEPPIC) an alkyl poly-glycoside, 52% aqueous solution.
- 30 NaCl: Sodium chloride: electrolyte.
- NH4CL: Ammonium chloride: electrolyte.
- 35 GUM: JAGUAR C13-S (RTM): Guar hydroxy propyl tri-methyl ammonium chloride, 100% solid, available in the marketplace from Meyhall (Rhone-Poulenc).

PERF: Commercially available perfume.

PRES: Preservative (formalin):

5     EXAMPLES 1-12:

Compositions were prepared as given in Table 1 below, by  
mixing of the ingredients as specified, other than the  
SLES, in an aqueous mixture at room temperature and final  
10     addition of the SLES as a 70% paste, as supplied, also at  
room temperature. All additions were made under mild  
shear. All quantities are given in absolute terms  
accounting for water present in the raw materials, but not  
accounting for other impurities except as mentioned below.  
15     Comparative examples are indicated by an asterisk.

The composition of Example 5, containing the guar gum  
(GUM), was prepared as follows: 40%wt of the salt was  
mixed together with the CAPB, PRES and PERF at low shear.  
20     Separately, the balance of the water, balance of the salt,  
the GUM and a small quantity of alkali (0.03% KOH on  
product) were combined. In combining the second admixture  
it was considered important that the alkali was added to  
the water and salt prior to the GUM in order to prevent  
25     gelation of the GUM. The two pre-mixes were combined  
under low shear, and the SLES added as the final  
component.

Table 1

EXAMPLE												
	1*	2	3	4	5	6	7	8	9	10*	11	12
SLES	36	36	36	36	36	36	36	39	32	32	35	28
CAPB	9	9	9	9	9	9	9	6	8	8	57	
NaCl	2	3	4	5	5	6	8	7.5	7.5	10	8	7.5
GUM	-	-	-	-	1	-	-	-	-	-	-	-
PERF	2	2	2	2	2	2	2	2	2	2	2	2
PRES	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Water	-----to 100%-----											

The compositions of examples 1 and 10 did not exhibit suitable rheology. In Example 1, insufficient salt was believed to be present and the product was believed to form a viscous, near solid, possibly hexagonal phase.

5

In example 10, too much salt was present and the required lamellar phase was not formed. It was noted that the formulation of example 10 formed a lamellar phase in the absence of the perfume component.

10

The compositions of Examples 2-9 and 11-12 were pourable liquids which perceptibly thicken when used in a hand washing procedure (0.5-2 ml of product applied to the hands when wet).

15

EXAMPLES 13-20:

Compositions were prepared as given in Table 2 below, by mixing of the ingredients as specified, other than the SLES, in an aqueous mixture at room temperature and addition of the SLES as a 70% paste, as supplied, also at room temperature. All additions were made under mild shear. All quantities are given in absolute terms accounting for water present in the raw materials, but not accounting for other impurities. Comparative examples are again indicated by an asterisk.

20

25



TABLE 2

EXAMPLE								
	13	14	15	16	17	18	19	20*
SLES	40	40	48	40	48	48	40	40
SULP	10	-	-	-	-	-	-	-
ESUL	-	10	-	-	-	-	-	-
APG	-	-	12	10	-	-	-	-
NONA	-	-	-	-	12	12	-	-
NONB	-	-	-	-	-	-	10	10
NaCl	-	-	5	5	5	4	3	5
NH <sub>4</sub> Cl	5	4	-	-	-	-	-	-
PERF	2	2	2	2	2	2	2	2
PRES	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Water	-----to 100%-----							

25

The compositions of Example 20 did not exhibit suitable rheology as too much salt was believed present and the required lamellar phase was not formed.

30

The compositions of Examples 13-19 were pourable liquids which perceptibly thicken when used in a hand washing procedure (0.5-2 ml of product applied to the hands when wet).

CLAIMS

1. An aqueous, stable dilution-thickening, concentrated,  
personal washing composition comprising:
  - a) 20-60wt% surfactant other than soap or primary  
alcohol sulphate, and,
  - b) 1-20wt% electrolyte, the ratio of electrolyte to  
surfactant being sufficient to form a lamellar  
phase.
2. Composition according to claim 1 comprising 30-60wt%  
surfactant.
3. Composition according to claim 1 wherein the  
surfactant comprises 50-90% of at least one anionic  
surfactant.
4. Composition according to claim 3 wherein the at least  
one anionic surfactant comprises one or more of fatty  
acid ether sulphate, alkyl sulphosuccinate and fatty  
isethionate.
5. Composition according to claim 1 further comprising  
5-40%wt on surfactant of a synergistic mildness  
active selected from the group comprising  
polyethoxylated alcohols, polyethoxylated alkyl  
phenols, alkyl polyglycosides, sorbitan esters,  
polysorbates, alkanolamides, poloxamers, amine  
oxides, aminimides, betaines, amido betaines and  
sulpho betaines, sulphosuccinates and mixtures  
thereof, provided that the synergistic mildness  
active and the remaining surfactant do not both  
comprise the same compound.

6. Composition according to the claim 1 comprising:
- a) 30-60wt% surfactant, comprising a mixture of at least one betaine and at least one anionic surfactant other than soap or primary alcohol sulphate, in a ratio of 1:1 to 1:10, and,
  - b) 1-20wt% electrolyte, the ratio of electrolyte to surfactant being sufficient to form a lamellar phase.
7. Composition according to claim 6 wherein the at least one anionic surfactant other than soap or primary alcohol sulphate is a fatty acid ether sulphate salt.
8. Composition according to claim 1 further comprising at least one skin or hair conditioning agent.
9. Composition according to claim 8 comprising:
- a) 30-50wt% surfactant, comprising a mixture of at least one betaine and a fatty acid ether sulphate salt in a ratio of 1:1 to 1:10 and,
  - b) 3-10wt% electrolyte, the ratio of electrolyte to surfactant being sufficient to form a lamellar phase, and,
  - c) 0.1-5wt% guar hydroxy propyl tri-methyl ammonium chloride polymer having a cationic charge density greater than 0.0001 in aqueous solution.

## INTERNATIONAL SEARCH REPORT

International Application No.

PCT/EP 94/00146

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 5 A61K7/50

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
E	WO,A,94 03152 (UNILEVER PLC) 17 February 1994 see the whole document ---	1-9
E	WO,A,94 03146 (L'OREAL) 17 February 1994 see the whole document ---	1-8
P,X	WO,A,93 25650 (HENKEL KGAA) 23 December 1993 see the whole document ---	1-9
P,X	EP,A,0 572 776 (HÜLS AKTIENGESSELLSCHAFT) 8 December 1993 see the whole document ---	1-8
P,X	EP,A,0 542 526 (UNILEVER PLC.) 19 May 1993 see the whole document ---	1-5,8
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Patent family members are listed in annex.

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1 National Application No

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passage	Relevant to claim No.
X	EP,A,0 281 027 (HENKEL KGAA) 7 September 1988 see example 5 ---	1-5
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